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(54) Title: **MULTICOMPONENT COATING AND ADHESIVE MATERIAL**

[see source for English]

Multicomponent Coating and Adhesive Material

Description:

Reactive, solvent-free adhesives which crosslink with atmospheric oxygen, in particular hot-melt adhesives, are well known in the state of the art. Such adhesives are produced from low-molecular starting materials such as polyesters or polyethers which contain OH groups and are then reacted with a stoichiometric excess of isocyanates for form reactive isocyanate-terminated adhesives. Examples of the isocyanate component that may be used include methylene diphenyl 4,4'-diisocyanate (MDI), naphthalene 1,5-diisocyanate (NDI), hexane 1,6-diisocyanate (HDI), toluene 2,4-diisocyanate (TDI), 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethyl-cyclohexane [isophorone diisocyanate] (IPDI) or their prepolymers.

As an alternative, the reactive coating and adhesive materials described here may also be produced on the basis of silane-functionalized polymers. The addition of catalysts, resins and other additives and auxiliary agents is also conventional.

Depending on the above starting materials and their average molecular weights as well as the desired properties of the adhesive to be produced, the fundamental polyaddition reaction may take several hours. Two or more adhesive components are brought together and are processed under the influence of heat to form a hot molten mass which can be used in this condition as a hot-melt adhesive. The crosslinking reaction takes place with the uptake of moisture from the air and/or from a wetted surface. This means that after production, the hot-melt adhesive must be stored dry in the absence of atmospheric humidity.

For shipping and storage, such an adhesive is therefore packed in tightly sealable containers, e.g., tin drums with a capacity of 20 or 200 L, where they are stored dry and in the absence of moisture. Before use, the cooled adhesive, which is usually in the form of a solid, is melted out of these containers and sent to the processing sites and applicator units. The melting is accomplished, e.g., by means of a drum melter, in which the heated ram, equipped with sealing rings, is introduced under pressure into a container filled with

adhesive. By means of a gear pump installed in the ram, the molten hot-melt adhesive, which has a viscous to liquid consistency, is conveyed through heated tubing to an applicator unit. The melting capacity of such a drum melter is on the order of 20-80 kg/hour, depending on the size of the container and the adhesive formulation.

This melting capacity is not adequate for some applications, in particular for coating large areas. In addition, when using relatively small drums, almost continuous operation is impossible because of the need to frequently replace drums. Acquisition of a drum melter by the user of the hot-melt adhesive also constitutes a not insignificant investment. If the containers holding the hot-melt adhesive develops a leak during shipping, the adhesive may rapidly become unusable, in particular if exposed to moisture. Another disadvantage is that after opening the container the adhesive must be processed completely and relatively promptly because such adhesive residues rapidly become unusable.

Another disadvantage of the state of the art described above is the restriction that use of such systems is limited exclusively to reactive adhesives. Alternating the processing of reactive adhesives and thermoplastic adhesives (based on EVA, APAO or PA) is a disadvantage for economic reasons as well as for technical reasons.

Use of multicomponent systems in the production of hot-melt adhesives and coating substances is essentially known. The systems disclosed in European Patent 0 304 026 B1, in the *Handbook of Adhesives*, 2nd edition, pp. 581 ff and the *ICI Polyurethanes Book*, 2nd edition, pp. 93 ff. all have in common the fact that the components react only with one another and after the reaction is concluded they no longer have any remaining reactivity for follow-up reactions, e.g., with moisture. Again, after the components are mixed they must necessarily be sent directly for further use.

The technical problem defined here is solved by the objects of Claims 1 and 2, whereby it is first assumed according to Claim 1 that there is continuous production of the isocyanate-terminated coating and adhesive material according to this invention, e.g., directly at the site of its use by combining the individual components, heating them in particular with further mixing until achieving a liquid state in which the material is then available as a reactive coating and adhesive material. In addition, with the isocyanate-terminated coating and adhesive material, at least one component comprises at least one high-molecular reactive starting polymer and a second component comprises a reactive isocyanate-terminated crosslinking agent.

In the silane-functionalized coating and adhesive material according to Claim 2, the first component comprises a high-molecular starting polymer and a second component comprises a reactive silane-functionalized crosslinking agent.

In the method according to this invention, to produce a two-component or multicomponent hot-melt adhesive, a first adhesive component and at least one second adhesive component are introduced into a screw conveyor, for example, for mixing and heating. The components are melted and blended there, resulting in a coating substance and/or an adhesive can be processed further immediately and in particular can be sent directly for use, preferably for coating, by using suitable process parameters such as temperature, pressure, screw length, etc., depending on the desired coating or adhesive and the composition of the components.

In contrast with the known process in which the site of production and the site of use of the adhesive are different, and thus shipping with all the disadvantages described above is necessary, production of the coating and adhesive according to this invention take place on-site at the user's place, which may have a suitable screw conveyor, for example, which is both economically advantageous and technologically advantageous. The amount of adhesive actually needed can be produced in a controlled manner and this may be accomplished in a continuous process. The disadvantages of shipping or the disadvantages that occur after opening the containers according to the state of the art described here thus do not occur with this invention.

According to a first variant of this invention, isocyanate termination of the adhesive is provided, in particular a termination in which the first component comprises at least one higher molecular reactive starting polymer and the second component comprises a reactive isocyanate-terminated crosslinking agent.

According to a second variant of this invention, a silane functionalization of the adhesive is provided, in particular such that the second component comprises a silane-functionalized crosslinking agent.

The first and second adhesive components may be stored separately, thus yielding problem-free storage and shipping options accordingly as well as long storage times. According to one embodiment of this invention, both components may advantageously be in the form of granules, but essentially liquid storage or shipping forms are also possible.

Furthermore, sufficiently high melt outputs of more than 100 kg per hour and/or large supply quantities of coating material and/or adhesive material can easily be produced with a screw conveyor. Such a screw conveyor is known essentially as a single screw extruder, for example.

All the settings on the screw conveyor such as temperature, pressure, throughput time, etc., are determined and adjusted as a function of the coating material and adhesive material to be produced and thus as a function of the compositions of the components. The pressure prevailing along the conveyor zone should be sufficient to heat the components, but additional heat sources may also be provided so that temperatures of about 200°C can easily be achieved on a short conveyance distance.

A line, optionally heatable, can also be connected to the end of the conveyance zone of the screw conveyor, carrying the hot-melt adhesive thus produced in liquid form directly to the processing site. The coating material and/or adhesive material is produced according to this invention in an advantageous manner only when there is a corresponding demand. After using the coating material and/or adhesive material, it is simple to clean all the equipment that has come in contact with the inventive material because only one of the adhesive components, in particular the adhesive component that is free of crosslinking agent, is carried through the screw conveyor, the corresponding lines and other installations, thus achieving a cleaning and rinsing effect.

In addition, it may prove expedient to supply the adhesive which is produced in a comparatively large volume to processing via intermediate containers. This makes it possible to achieve a buffering function in the production of the adhesive and/or an adaptation to local specifications.

In this invention, the first adhesive component may be at least a polymer that is reactive in particular with respect to isocyanates and/or a resin component plus optionally a non-reactive polymer. Use of polymers that are reactive with isocyanate in particular and that have molecular weights of more than 8,000 g/mol has proven to be especially advantageous. Such higher molecular components, in particular those with an average molecular weight of 10,000 to 30,000 g/mol, e.g., polyesters, polycaprolactone polyesters, polyethers, polyurethanes, polyamides or polytetrahydrofurans which have at least two reactive groups per molecule with reactive hydrogen atoms are capable of

reacting with isocyanates. The use of such reactive polymers in amounts of 20-100 wt%, in particular in amounts of 50-95 wt% in the first adhesive component is also preferred.

The resin, e.g., aliphatic, cyclic or cycloaliphatic hydrocarbon resins, terpene-phenol resins, coumarone-indene resins, α-methylstyrene resins, polymerized tall resin esters or ketone-aldehyde resins which may be used in the first adhesive component are not limited in particular. Resins with a low acid value, in particular with values lower than 1 mg KOH/g are preferred for use, however. The amounts of the resin(s) in the first adhesive component may be preferably 5 to 35 wt%, for example, and essentially between 0 and 70 wt%.

The first adhesive component may also contain a non-reactive polymer, whereby when using ethylene/vinyl acetate copolymers, such polymers having a vinyl acetate content of 12% to 40%, in particular 18% to 28% and a melt index (DIN 53735) of 8 to 800, in particular from 150 to 500, are preferred. However, it may also be advantageous to use polyolefins. Polyolefins with average molecular weights M_n of 5,000 to 25,000 g/mol, in particular 10,000 to 20,000 g/mol and with softening ranges according to the ring and ball method between 80°C and 170°C, in particular between 80°C and 130°C can be used to advantage in the method according to this invention. The amounts of the non-reactive polymer(s) in the first adhesive component are not particularly critical and are selected according to the desired coating material and/or adhesive material. For example, 50 wt% to 35 wt% non-reactive polymer may be present in the first adhesive component.

In the first variant of the adhesive according to this invention, the second adhesive component comprises at least one reactive crosslinking agent component which may comprise at least one isocyanate that is solid at room temperature, or alternatively in the second variant with a silane-functionalized adhesive, it may be a silanized polymer that is solid at room temperature.

The following isocyanate-terminated crosslinking agents have proven to be especially advantageous: methylene diphenyl 4,4'-diisocyanate (MDI), triphenylmethane 4,4',4"-triisocyanate, tris(4-isocyanatophenyl) thiophosphate, naphthalene 1,5-diisocyanate (NDI) or isomers thereof, dimers of toluene 2,4-diisocyanate (TDI) and 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethyl-cyclohexane [isophorone diisocyanate] (IPDI) or their hydrogenation products and trimers of 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethyl-cyclohexane [isophorone diisocyanate] (IPDI).

In this invention, adhesive components may also contain various other additives which are fundamentally known for use in reactive adhesives such as plasticizers, e.g., plasticizers based on phthalic acid or phosphate ester compounds, glycol acetate, high boiling organic oils, esters or other additives that are helpful in plastification, stabilizers, antioxidants, acid scavengers and/or anti-aging agents. These optional components of the adhesive are selected according to the intended use of the finished adhesive. This is a procedure which is known for those skilled in the art in this field.

The coating materials and/or adhesives according to this invention produced by the method according to this invention are suitable in particular for bonding foam upholstery, upholstered furniture and mattresses or may be used for continuous coating purposes such as sheathing profiles. They may be applied with known methods, e.g., by spray application, injection application, nozzle application or roller application.

Exemplary embodiments of the method according to this invention and the adhesive components to be used in the process are explained in greater detail below.

Example 1

Isocyanate-terminated adhesive

A first adhesive component was prepared in the form of thermoplastic granules having the following composition:

- polyester with average molecular weight of approx. 20,000 g/mol and an OH number of 5: 60 wt%
- ethylene/vinyl acetate copolymer with a VA content of 18% and a melt index of approx. 150: 20 wt%
- low-molecular aromatic hydrocarbon resin with a softening range of 75-85°C and an acid number of less than 1 mg KOH/g: 20 wt%

A second adhesive component in the form of thermoplastic granules having the following composition was produced:

Polyisocyanate based on cycloaliphatic 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethyl-cyclohexane [isophorone diisocyanate] (IPDI) having an isocyanate content of 17% and a functionality between 3 and 4.

The first adhesive component was supplied through a first feed mechanism of a single-screw extruder heated to 200°C of the conveyor worm gear of the extruder, while the second adhesive component was fed by metered addition through a second feed mechanism at a mixing ratio of 7:1. The melting capacity was 120 kg/hour.

An isocyanate-terminated reactive hot-melt adhesive was obtained with which it was possible to produce profiles with a veneer, decorative paper film or plastic film and a core made of MDF or particle board on known profile sheathing installations.

Example 2

Silane-functionalized adhesive

A first adhesive component in the form of thermoplastic granules having the following composition was produced:

31.2 wt% polyolefins
(Vestoplast 708, Degussa Hüls AG)
62.4 wt% hydrocarbon resin
(Escorez 5320, Exxon Deutschland GmbH)
6.2 wt% wax
(Paraflint)
0.2 wt% dibutyltin dilaurate catalyst
(DBTL from the Huntsmann Corp.)

A second adhesive component in the form of thermoplastic granules having the following composition was produced:

95 wt% silanized polyolefins
(Vestoplast 206V, Degussa Hüls AG)
5 wt% PE wax
(Polywax 1000)

The first adhesive component was supplied to the delivery screw of the extruder through a first feed mechanism of a single screw extruder heated to 200°C, while the second adhesive component was supplied in a metered manner through a second feed mechanism at a mixing ratio of 1:1.9. The melting capacity was 120 kg/hour.

A silane-functionalized reactive hot-melt adhesive was obtained with which it was possible to produce profiles consisting of a veneer, decorative paper film or plastic film with a core of MDF or particle board on the known profile sheathing installations.

Multicomponent Coating and Adhesive Material

Patent Claims:

1. Multicomponent isocyanate-terminated coating and adhesive material, characterized by continuous production by blending the individual components, heating them, in particular while continuing to blend until they reach a liquid state in which the coating and adhesive material, which is then reactive, is supplied to profile sheathing installations, coating installations and the like for use there, in particular being supplied to them directly, whereby one component comprises a high-molecular reactive starting polymer and a second component comprises a reactive isocyanate-terminated crosslinking agent.

2. Multicomponent silane-functionalized coating and adhesive material characterized by a continuous production by blending the individual components, heating them, in particular while continuing to blend until they reach a liquid state in which the coating and adhesive material, which is then reactive, is supplied to profile sheathing installations, coating installations and the like, in particular being supplied to them directly, whereby one component comprises a higher molecular starting polymer, and a second component comprises a reactive silane-functionalized crosslinking agent.

3. Coating and adhesive material according to one or more of the preceding claims, characterized in that at least one adhesive component is in the form of granules.

4. Coating and adhesive material according to one or more of the preceding claims, characterized in that the starting polymer which is reactive in particular with isocyanates has an average molecular weight between 8,000 and 50,000 g/mol, in particular between 10,000 and 30,000 g/mol.

5. Coating and adhesive material according to one or more of the preceding claims, characterized in that the polymers that are reactive in particular with isocyanates

are selected from the group consisting of polyesters, polycaprolactone polyesters, polyethers, polyurethanes, polyamides and/or polytetrahydrofurans.

6. Coating and adhesive material according to one or more of the preceding claims, characterized in that the amount of polymers that are reactive with isocyanates in particular in the first adhesive component constitutes 20 to 100 wt%, in particular 50 to 95 wt%.

7. Coating and adhesive material according to one or more of the preceding claims, characterized in that one or more adhesive components contain at least one resin.

8. Coating and adhesive material according to Claim 7, characterized in that resins are selected from the group of aliphatic, cyclic or cycloaliphatic hydrocarbon resins, terpene-phenol resins, coumarone-indene resins, α-methylstyrene resins, polymerized tall resin esters and/or ketone-aldehyde resins.

9. Coating and adhesive material according to one or more of Claims 7 through 9, characterized in that the amount of resin in one or more adhesive components amounts to 0 to 70 wt%.

10. Coating and adhesive material according to one or more of the preceding claims, characterized in that the first adhesive component comprises a non-reactive polymer.

11. Coating and adhesive material according to Claim 10, characterized in that non-reactive polymers are selected from the group of ethylene/vinyl acetate copolymers and/or polyolefins.

12. Coating and adhesive material according to Claim 10 or 11, characterized in that the ethylene/vinyl acetate copolymers have a vinyl acetate content between 12% and 40%, in particular between 18% and 28% and have a melt index between 8 and 800, in particular between 150 and 500.

13. Coating and adhesive material according to one or more of Claims 10 through 12, characterized in that the polyolefins have an average molecular weight M_n between 5,000 and 25,000 g/mol, in particular between 10,000 and 20,000 g/mol,

and have a softening range between 80 and 170°C, in particular between 80 and 130°C.

14. Isocyanate-terminated coating and adhesive material according to one or more of the preceding claims, characterized in that the reactive crosslinking agent component comprises an isocyanate that is solid at room temperature.

15. Coating and adhesive material according to Claim 14, characterized in that the solid isocyanates are selected from the group consisting of methylene diphenyl 4,4'-diisocyanate (MDI), triphenylmethane 4,4',4"-triisocyanate, tris(4-isocyanatophenyl) thiophosphate, naphthalene 1,5-diisocyanate (NDI) or isomers thereof, dimers of toluene 2,4-diisocyanate (TDI) and 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethyl-cyclohexane [isophorone diisocyanate] (IPDI) or their hydrogenation products and trimers of 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethyl-cyclohexane [isophorone diisocyanate] (IPDI).

16. Silane-terminated coating and adhesive material according to one or more of the preceding claims, characterized in that the reactive crosslinking agent component comprises at least one silanized polymer that is solid at room temperature.

17. Coating and adhesive material according to one or more of the preceding claims, characterized in that the mixing ratio of the first and second adhesive components is between 20:1 and 1:20.